Surgical procedures require instruments that can withstand aggressive environments within the human body and during the sterilization processes. Due to its high corrosion and wear resistance as well as antibacterial properties, diamond-like carbon (DLC) has become the material of choice for protective coatings in the biomedical sector. DLC with high sp³ content can be produced by plasma assisted chemical vapor deposition (PACVD); however, synthesis conditions must be optimized to yield DLC films with the desired properties.

**Background**
- DLC is a type of amorphous carbon where properties are dependent on the composition and bonding type.
- Synthesis conditions can be adjusted to control sp³ content, during PACVD.
- DLC coatings are considered “acceptable” if sp³ carbon exceeds 30%, resistivity reaches >1GΩ without dielectric breakdown, and contains a microhardness >5 GPa

**Objective**
The project is aimed to optimize PACVD process in order to obtain high quality DLC coatings.

**Experimental Procedure**

**Material Synthesis**
PACVD (Rübiger PN 70/90)
- Plasma decomposes precursor gases, and reactants form a solid state coating on substrate.

**Material Characterization**
- Raman Spectroscopy (Thermo Scientific DXR Raman Microscope)
  - for sp³ content
  - Raman probes sp² and sp³ molecular vibrations that can be used to estimate composition
  - Raman data calibrated with an Si wafer, and collected at 50x magnification, using a 532 nm laser wavelength
  - Scanning Electron Microscopy, SEM (Phenom Desktop)
  - for microstructure
  - Cross sectional views estimate coating thickness while surface images provide qualitative insight to coating porosity and density
  - Micrographs taken at 5,000x magnification, with 15 kV accelerating voltage

**Indentation Testing** (Hysitron Tri 950 Triboindenter)
- for microhardness
  - All indents made with Berkovich tip and 5 second incremental load-hold-unload cycle

**High Potential Testing, HiPot** (Megger MIT 330)
- for resistivity
  - High potential (HiPot) tests conducted with an applied voltage of 1 kV
  - Sample passes test as nonconductive, if the measured current is greater than 1 GΩ, and consistent along the sample

**Results**

**Design of Experiment, DoE**
- Samples coated on steel substrate
- Rubig PN 70/90 contained inherent parameter independence
- Multiple synthesis parameters were varied simultaneously

**Microstructure**
- Micrographs of DLC thickness overlaid on microstructure
- Sample 1 contained smaller grains

**Microhardness**
- Load (μN)
- Depth (nm)
- Sample 1
- Sample 2

**Resistivity**
- Sample 1
- Resistivity (MO)
- Pass
- 1 0.03 NO
- 2 0.02 NO

**Raman Spectra**
- Indicators of sp³ content rely on:
  - G band peak position
  - D and G band intensity ratios
- The G band peak positions were too similar to distinguish between samples
- The D/G band intensity ratio showed Sample 1 containing more sp³ carbon
- Both samples provided a qualitative range of sp³ content (30-40%) for the PACVD synthesis parameters

**Discussion**
- While a single characterization technique could not distinguish samples, all techniques qualitatively indicated Sample 1 contained a higher sp³ content
- How did synthesis parameters influence sp³ content?
  - Gas ratio → Increasing C₂H₂ gas should form more sp³ due to more carbon (less hydrogen) involved in the synthesis
  - Pressure → Higher operating pressure should form a denser coating due to an increase in the number of effective collisions during synthesis
  - Process Time → A longer process time would extend overall exposure of reactants to substrate, increasing coating thickness
- Operating power → A higher operating power should increase ion energy during, increasing sp³ content
- Due to limited sample size and the parameter interdependence, quantitative conclusions were not found.

**Conclusion**
Material synthesis of a DLC coating relies on the understanding of PACVD instrument parameters. Based on the sample size, characterization techniques only began to distinguish the relationship between operating conditions and coating quality. Sample 1 surpassed sample 2 in hardness, resistivity, and sp³ content. These qualities were most likely attributed to higher operating pressure and power. The differences that did exist were not significantly different but does not exclude the prevalence of these trends.

**Recommendations**
To increase sp³ content, the relationship between the synthesis parameters and coating properties must be fully understood. A larger sample size should be used to independently test all parameters of the PACVD instrument.

**References**


MSE 430-440: Materials Processing and Design